IS 8232 (1976): 2,6-Diaminoanthraquinone, Technical [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]

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IS : 8232 - 1976

Indian Standard
SPECIFICATION FOR 2,6-DIAMINOANTHRAQUINONE, TECHNICAL

UDC 667·282·14 : 547·673·5

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

February 1977

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TO

IS:8232-1976 SPECIFICATION FOR
2,6-DIAMINOANTHRAQUINONE,
TECHNICAL

[*Page 4, Table 1, Sl No. (i), col 3*] - Substitute '90.0' *for* '88.0'.

(FCDC 11)

Reprography Unit, ISI, New Delhi, India

Indian Standard

SPECIFICATION FOR 2,6-DIAMINOANTHRAQUINONE, TECHNICAL

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(*Continued on page* 2)

(*Continued from page* 1)

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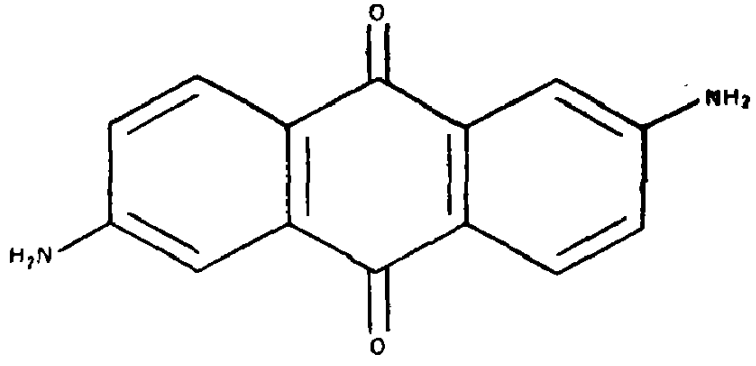
Indian Standard

SPECIFICATION FOR 2,6-DIAMINOANTHRAQUINONE, TECHNICAL

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 25 August 1976, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Chemical Division Council.

0.2 2,6-Diaminoanthraquinone ($C_{14}H_{10}O_2N_2$) is an important dye intermediate, used mainly in the manufacture of vat dyes. It has the following structural formula:



2, 6-DIAMINOANTHRAQUINONE

(Molecular Mass 238·2)

0.3 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes the requirements and the methods of sampling and test for 2, 6-diaminoanthraquinone, technical.

*Rules for rounding off numerical values (*revised*).

2. REQUIREMENTS

2.1 Description — The material shall be in the form of dark, reddish-brown powder and shall be free from visible impurities.

2.2 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR 2, 6-DIAMINOANTHRAQUINONE, TECHNICAL

Sl No.	Characteristic	Requirement	Method of Test, Ref to Cl No. in	
			Appendix A	IS : 5299-1969*
(1)	(2)	(3)	(4)	(5)
i)	Assay, percent by mass (on dry basis), *Min*	88 0	A-2	—
ii)	2-Aminoanthraquinone, percent by mass (on dry basis), *Max*	2 0	A-3	—
iii)	Moisture, percent by mass, *Max*	0 5	—	9·3
iv)	Ash, percent by mass (on dry basis), *Max*	2·0	—	11·1
v)	Iron content (as Fe), percent by mass (on dry basis), *Max*	0·2	A-4	—

*Methods of sampling and tests for dye intermediates.

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in steel drums (*see* IS : 2552-1970*) lined with suitable polyethylene film or as agreed to between the purchaser and the supplier. The containers shall be securely closed.

3.2 Marking — Each container shall bear legibly and indelibly the following information:

a) Name of the material;

b) Name of the manufacturer and his recognized trade-mark, if any;

c) Batch number; and

d) Tare, net mass and gross mass

*Specification for steel drums (galvanized and ungalvanized) (*first revision*).

3.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — *The use of the ISI Certification Mark is governed by the provisions of the* Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in 3 of IS : 5299-1969*.

4.2 Number of Tests

4.2.1 Test for assay and 2-aminoanthraquinone and shall be conducted on each of the individual samples.

4.2.2 Tests for the remaining characteristics, namely moisture, ash, and iron content shall be conducted on the composite sample

4.3 Criteria for Conformity

4.3.1 *For Individual Samples* — The lot shall be declared as conforming to the requirements of assay and 2-aminoanthraquinone if each of the individual test results satisfies the relevant requirement given in Table 1.

4.3.2 *For Composite Sample* — For declaring the conformity of the lot to *the requirements of the characteristics tested on the composite sample* (*see* **4.2.2**), the test results for each of the characteristics shall satisfy the relevant requirement given in Table 1.

5. TEST METHODS

5.1 Tests shall be carried out according to the methods prescribed in Appendix A and IS : 5299-1969*, as indicated in col 4 and 5 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1960†) shall be employed in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

*Methods of sampling and tests for dye intermediates.

†Specification for water, distilled quality (*revised*).

APPENDIX A

(*Table* 1 and *Clause* 5.1)

METHODS OF TEST FOR 2, 6-DIAMINOANTHRAQUINONE, TECHNICAL

A-1. PREPARED SAMPLES

A-1.1 Dry the material at 105°C to constant mass Grind and mix well. Transfer the material to a wide-mouthed bottle and stopper it. Do not expose the sample to an atmosphere containing acidic or alkaline fumes. Use this *prepared sample* for test.

A-2. ASSAY

A-2.0 Outline of the Method — Purity of the material is estimated by chromatographic method. 2, 6-Diaminoanthraquinone is separated chromatographically and determined quantitatively by spectrophotometer.

NOTE — The solutions of 2, 6-diaminoanthraquinone are slightly sensitive to light and therefore should be stored in the dark before measuring optical density; and during chromatography, light shall be excluded from the column as much as possible.

A-2.1 Apparatus

A-2.1.1 *Chromatographic Column* — a glass tube, 40 cm long, having an internal diameter of 1·5 cm, joined with a 50-ml thistle funnel at the upper end and fitted with a stop-cock at the lower end. Set up the column vertically such that the percolation passing through the column can be collected in a 250-ml conical flask Place a cotton-wool plug in the tube and press it to the bottom by means of a glass rod, flattened latitudinally at the end Place a disc of filter paper cut to the approximate internal diameter of the chromatographic tube on the top of the cotton wool.

A-2.1.2 *Sintered Glass Crucible* — G No. 4

A-2.1.3 *Vacuum-Oven*

A-2.1.4 *Spectrophotometer*

A-2.2 Reagents

A-2.2.1 *Alumina* — aluminium oxide, standardized for chromatographic absorption analysis in accordance with the method given in **A-4.1** of IS : 5044-1969*.

*Specification for benzanthrone.

A-2.2.1.1 *Deactivation of standard alumina* — To 100 g of standard alumina add 3 to 5 ml of water, as required, to obtain effective separation. Mix thoroughly by shaking vigorously for 15 minutes.

A-2.2.2 *Pyridine* — pure, dried over fused calcium chloride, filtered and distilled before use Portion distilling between 115°C and 116°C shall be collected for use It shall be clear and colourless

A-2.2.3 *Toluene* — pure, dried over fused calcium chloride, filtered and distilled before use. Portion distilling between 110°C and 111°C shall be collected for use. It shall be clear and colourless

A-2.2.4 *Solvent Mixture A* — To 150 ml of toluene add 850 ml of pyridine and mix well.

A-2.2.5 *Solvent Mixture B* — Add 6·0 ml of water to 1000 ml of solvent mixture *A* and mix well

A-2.2.6 *Standard 2,6-Diaminoanthraquinone* — Dissolve 200 mg of the *prepared sample* (**A-1.1**) in 200 ml of pyridine Reflux for half an hour. Cool to room temperature and transfer to a 250-ml volumetric flask. Add 37·5 ml of toluene and make up to the mark with pyridine

A-2.2.6.1 Transfer the solution obtained as in **A-2.2.6** gradually to the chromatographic tube prepared as prescribed in **A-2.3.1** When all the solution loaded has entered the column and the cotton wool at the top of the alumina is just dry add the solvent mixture *A* in small lots until the bands start moving down the column Continue elution with solvent mixture *A* until there is distinct separation of the bands, which will be in the following order from bottom to top

Faint yellow	2-Aminoanthraquinone
Orange main band	2, 6-Diaminoanthraquinone
Faint purple	Impurity
Strongly adsorbed band	At the top

A-2.2.6.2 Elute with solvent mixture *A* until the faint yellow band of 2-aminoanthraquinone leaves the column Discard the fraction. Now fill the column with solvent mixture *B* and continue elution when the main orange band of 2, 6-diaminoanthraquinone starts moving down the column.

A-2.2.6.3 Collect the main orange-coloured eluate which represents pure 2, 6-diaminoanthraquinone A battery of five to six tubes is kept in operation for collecting a sizeable quantity of the material Filter through filter paper to remove any stray alumina particles Concentrate the total quantity of eluate of 2, 6-diaminoanthraquinone by distillation of the eluent until crystallization occurs on cooling to room temperature. The dry pulverized pure product is further boiled in a beaker with water for 15 minutes to remove the solvent of crystallization. Cool and filter on a sintered glass crucible Wash and dry in a vacuum oven at 100°C.

A-2.2.6.4 In order to check its purity, dissolve about 10 mg, accurately weighed, in 10 ml of pyridine and pass the cooled solution through a chromatographic column set up as prescribed in **A-2.3.1**, eluting with solvent mixture *B* as necessary. Collect the band carefully (only one band should appear), and determine its optical density at 394 nm. Make up a solution of purified crystals using the same proportions of crystals to solvent. Determine its optical density in the same way. A difference in the two figures for optical density amounting to more than 0·003 indicates the presence of an impurity. The chromatographic purification shall in such a case be repeated until this check test is satisfied.

A-2.3 Procedure

A-2.3.1 *Preparation of Chromatographic Tube* — Prepare a slurry of about 15 g of deactivated alumina in solvent mixture *A* and pour it into the tube. Wash down the sides of the tube and pack the column by lightly tapping the tube. Place, first a disc of filter paper and then a cotton-wool plug at the top of the alumina column. Care shall be taken to see that the alumina column remains soaked with the solvent, such that at least a 2-cm layer of the solvent remains always on top of the alumina On no account shall the alumina be left to dry. In the event of the latter happening, reslurry the alumina in the tube and repack.

A-2.3.2 *Preparation of Standard Calibration Graph for Particular Optical Instrument* — Prepare a number of standard solutions of chromatographically pure 2,6-diaminoanthraquinone obtained as in **A-2.2.6** in solvent mixutre *B*, varying in concentration from 0·6 mg/100 ml to 2·0 mg/100 mg with a difference of 0·2 mg/100 ml between successive concentrations. Take readings for optical density or percentage transmittance for these concentrations at a wavelength of 394 nm (*see* Notes 1 and 2) using specially matched cells specified for the spectrophotometer used. The temperature of the solutions immediately before and after measurements shall be 27 ± 2°C. Where the instrument is calibrated in percentage transmittance only, the optical density may be read out from a standard table. Plot the calibration curve of concentration (in mg/100 ml) against optical density.

NOTE 1 — Maximum absorption (or minimum transmittance) has been observed at a wavelength of 394 nm by standard instruments However, this may need checking with the particular instrument to be used. Several readings for absorption using solutions of varying concentrations are taken at different wavelengths and the one for maximum absorption determined from the graph.

NOTE 2 — The standard calibration curve and the spectrophotometer shall be checked for accuracy periodically.

A-2.3.3 *Determination of the Purity of the Sample*

A-2.3.3.1 Weigh accurately about 35·0 mg of the *prepared sample* (**A-1.1**) in a 250-ml conical flask. Add about 200 ml of pyridine. Reflux

for half an hour using a condenser. Cool to room temperature by keeping in a water-bath. Transfer to a dry 250-ml volumetric flask. Rinse the conical flask with small amounts of pyridine two to three times and transfer the rinsings also to the volumetric flask. Add 37·5 ml of toluene, dilute to the mark with pyridine and shake well (solution 1).

A-2.3.3.2 Prepare a solvent-soaked alumina column using a chromatographic tube of 40 cm length and 1·5 cm internal diameter, with deactivated alumina as adsorbent and solvent mixture *A* as solvent. Pack the column as in **A-2.3.1**.

A-2.3.3.3 Load 10 ml out of the 250 ml of solution 1 of 2, 6-diaminoanthraquinone. When all the solution has entered the column and cotton wool at the top of the alumina is just dry, add a small quantity of solvent mixture *A*. Go on adding this solvent in small lots till cotton wool on top of the alumina column is colourless and the reddish-orange band of pure 2, 6-diaminoanthraquinone just leaves the top of the alumina column. Elute until the faint yellow band of 2-aminoanthraquinone leaves the column. Collect the faint yellow band of 2-diaminoanthraquinone for test in **A-3**. Now fill up the column with solvent mixture *B* and continue elution.

A-2.3.3.4 Collect the orange eluate of 2, 6-diaminoanthraquinone in a dry 100-ml volumetric flask and make up to the mark with solvent mixture *B*. Shake the contents thoroughly to give solution 2. Use this solution to take readings for either percentage transmittance or absorption using the particular spectrophotometer.

A-2.3.3.5 Adjust the wavelength of maximum absorption at 394 nm, and then adjust the instrument in such a way that the transmittance through the blank is recorded at the 100 percent reading after inserting the cell with the blank solution solvent mixture *B*. Now replace the cell with solution 2 of the sample and read percentage transmittance. Referring to the standard tables for conversion of transmittance to optical density read out the concentration *C* against the optical density from the standard calibration graph (**A-2.3.2**) in terms of milligrams per 100 millilitres of the final dilute solution 2.

A-2.4 Calculation

$$\text{Assay, percent by mass} = \frac{C \times 2500}{M}$$

where

C = mass in mg of 2,6-diaminoanthraquinone contained in 100 ml of solution 2, and

M = mass in mg of the material taken for the test.

A-3. DETERMINATION OF 2-AMINOANTHRAQUINONE

A-3.0 Outline of the Method — 2-Aminoanthraquinone is separated chromatographically and determined quantitatively by spectrophotometer.

A-3.1 Reagents

A-3.1.1 *2-Aminoanthraquinone* — chromatographically pure.

A-3.2 Procedure

A-3.2.1 Collect the faint yellow band of 2-aminoanthraquinone (*see* **A-2.3.3.3**) in a 100-ml flask and read in 1-cm cell at a wavelength of 435 nm against a blank of solvent mixture *A* (**A-2.2.4**).

A-3.2.2 Weigh 0·1 g of chromatographically purified 2-aminoanthraquinone in 200 ml of solvent mixture *A* (**A-2.2.4**). Reflux for 15 minutes, cool and transfer to a 250-ml volumetric flask. Make up to the mark with solvent mixture *A*. Pipette 10 ml of this solution in a 100-ml volumetric flask and make up to the mark with solvent mixture *A*. Read this solution in 1-cm cell at a wavelength of 435 nm.

A-3.3 Calculation — Calculate the percentage of 2-aminoanthraquinone by comparing the optical density obtained as in **A-3.2.1** with that of the chromatographically purified standard of 2-aminoanthraquinone obtained as in **A-3.2.2**, using the following formula :

$$\text{2-Aminoanthraquinone, percent by mass} = \frac{D_1 \times M}{D \times M_1} \times 100$$

where

D_1 = optical density of the material under test,
M = mass in g of the standard,
D = optical density of the standard, and
M_1 = mass in g of the material taken for the test.

A-4. DETERMINATION OF IRON CONTENT

A-4.0 Outline of the Method — Thioglycolic acid gives a reddish-violet colouration with ferrous and ferric ions in an alkaline medium. The residue from the ash determination is dissolved in hydrochloric acid, thioglycolic acid added and the solution then made alkaline by the addition of ammonia. The colour developed is compared with that obtained with standard iron solution similarly treated.

A-4.1 Apparatus

A-4.1.1 *Volumetric Flasks* — One of 25 ml capacity and two of 1 000 ml capacity.

A-4.1.2 *Nessler Cylinders* — two of 100 ml capacity.

A-4.2 Reagents

A-4.2.1 *Hydrochloric Acid* — relative density 1·16.

A-4.2.2 *Ammonium Hydroxide* — relative density 0·90.

A-4.2.3 *Thioglycolic Acid Solution* — 10 percent (*v/v*).

A-4.2.4 *Citric Acid Solution* — 30 percent (*m/v*).

A-4.2.5 *Standard Iron Solution* — Dissolve 0·702 2 g of ammonium ferrous sulphate [$(NH_4)_2SO_4 . FeSO_4 . 6H_2O$] in 50 ml of dilute sulphuric acid and transfer to one of the 1 000-ml volumetric flasks. Dilute with water to 1 000 ml and mix well. Pipette out 100 ml of this solution into the second 1 000-ml volumetric flask. Dilute again to 1 000 ml. One millilitre of this solution contains 10 μg of iron.

A-4.3 Procedure — To the platinum or silica basin containing the residue from the determination of ash [Table 1, Sl No. (iv)] add 5 ml of hydrochloric acid. Heat the basin on a boiling water-bath, agitating with a stirrer of platinum wire, until all the residue has dissolved. Allow to cool, transfer to the 25-ml volumetric flask, dilute to the mark with water and mix thoroughly. For each determination transfer 10 ml of this solution to a 100-ml Nessler cylinder, dilute to about 30 ml and add 0·5 ml of citric acid solution followed by 1 ml of thioglycolic acid solution. Add ammonium hydroxide carefully until a reddish-purple colour just appears and then add 0·5 ml in excess. Dilute to 100 ml and mix thoroughly. To about 90 ml of water in the second 100-ml Nessler cylinder add 2 ml of hydrochloric acid and 0·5 ml of citric acid solution followed by 1 ml of thioglycolic acid solution and 3 ml of ammonium hydroxide. Add standard iron solution slowly from a burette shaking with each addition until the depth of colour in the two Nessler cylinders is identical when they are viewed along their axis. Record the volume of standard iron solution added.

A-4.4 Calculation

$$\text{Iron content (as Fe), percent by mass} = \frac{V \times 0{\cdot}002\,5}{M}$$

where

V = volume in ml of standard iron solution used, and

M = mass in g of the material taken for the determination of ash.

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

Quantity	*Unit*	*Symbol*
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic *temperature*	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

Quantity	*Unit*	*Symbol*
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

Quantity	*Unit*	*Symbol*	*Conversion*
Force	newton	N	1 N = 1 kg.1 m/s^2
Energy	*joule*	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m^2
Frequency	hertz	Hz	1 Hz = 1 c/s (s^{-1})
Electric conductance	*siemens*	S	1 S = 1 A/V
Pressure, stress	pascal	Pa	1 Pa = 1 N/m^2

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